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(54) DISPOSITIF DE RETENTION DE VAISSEAU DE REACTION
(54) REACTION VESSEL HOLDING DEVICE

(57)
The invention relates to a reaction vessel holder, comprising a fixed piece (1) and a plurality of couplings (4) fixed thereto. Reaction vessels (5) are fixed to these couplings (4). The couplings (4) and the fixed piece (1) have apertures through which a feed and/or withdrawal tool, in particular a needle, spoon or gripper, can be introduced into the reaction vessel (5). Part of each coupling (4) is flexible, so that the reaction vessels (5) fixed to the coupling (4) can be shaken by a shaking device (7) without moving the fixed piece (1).

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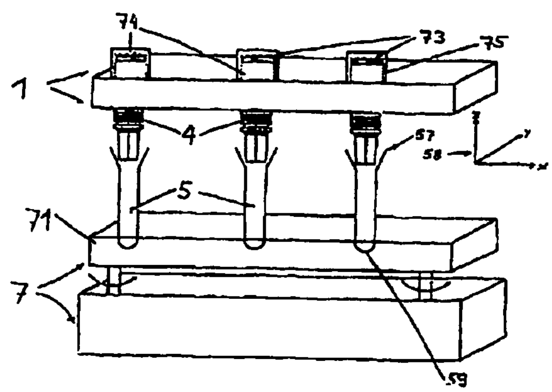
(54) **DISPOSITIF DE RETENUE D'EPROUVETTES**
(54) **REACTION VESSEL HOLDER**



(57) Ce dispositif de retenue d'éprouvettes comprend une partie fixe (1) et une pluralité de raccords (4) assujettis à celle-ci auxquels sont assujetties les éprouvettes (5). Les raccords (4) et la partie fixe (1) comprennent des ouvertures à travers lesquelles un outil d'amenée et/ou de prélèvement, en particulier une aiguille, une cuiller ou une pince, peut être introduit dans l'éprouvette (5). Une partie de chaque raccord (4) est flexible, ce qui permet à un secoueur (7) de secouer les éprouvettes (5) assujetties au raccord (4) sans faire bouger la partie fixe (1).

(57) The invention relates to a reaction vessel holder, comprising a fixed piece (1) and a plurality of couplings (4) fixed thereto. Reaction vessels (5) are fixed to these couplings (4). The couplings (4) and the fixed piece (1) have apertures through which a feed and/or withdrawal tool, in particular a needle, spoon or gripper, can be introduced into the reaction vessel (5). Part of each coupling (4) is flexible, so that the reaction vessels (5) fixed to the coupling (4) can be shaken by a shaking device (7) without moving the fixed piece (1).

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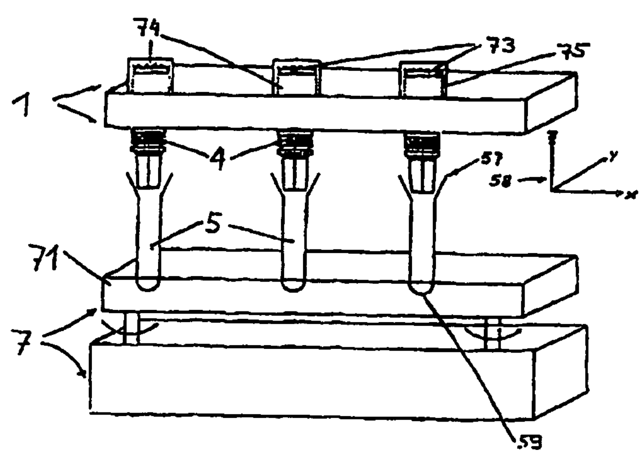

(57) Abstract

The invention relates to a reaction vessel holder, comprising a fixed piece (1) and a plurality of couplings (4) fixed thereto. Reaction vessels (5) are fixed to these couplings (4). The couplings (4) and the fixed piece (1) have apertures through which a feed and/or withdrawal tool, in particular a needle, spoon or gripper, can be introduced into the reaction vessel (5). Part of each coupling (4) is flexible, so that the reaction vessels (5) fixed to the coupling (4) can be shaken by a shaking device (7) without moving the fixed piece (1).

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Reaction Vessel Holding Device

The present invention relates to a reaction vessel holding device, as defined in the preamble of the independent Claim 1, and a reaction block with a reaction vessel holding device of this type.

In chemical research in the pharmaceutical industry and universities, it is becoming more and more important to discover as fast as possible a large number of potential active ingredients and then to test them. Some of the chemical research, therefore, currently deals with combinatory chemistry, parallel synthesis and high-speed chemistry. In this work, the possibility of being able to employ known or new types of chemical reactions with the least possible amount of adaptation and to the greatest extent is of central importance.

In consequence, the most varied types of devices have been created for carrying out a multiplicity of chemical, biochemical or physical processes in parallel. All of these processes, however, are either only suitable for special applications or are too complicated in construction, too large or are not user-friendly and/or do not permit the individual process steps to be sufficiently automated.

Such a device is marketed by Advanced Chemtech, Europe SA under the designation 496 MBS. This comprises a reaction block with a plurality of included depressions within which the reactions can be carried out. The complete block is moved by a vibration device, which is attached below it and this leads to a not very efficient mixing of the reactants in the depres-

sions. Liquids can be supplied to and/or extracted from the depressions by means of hollow needles, sealing of the individual depressions relative to the outside being realized by means of septa/septum plates.

This device also has the disadvantage that it is necessary to move the full weight in order to shake the reaction block. This is one reason why shaking (or vibration) is only carried out with small amplitudes. Shaking during the supply or extraction of fluid, which is often necessary or even essential, is impossible for technical reasons (needles would, for example, be moved along with the septum). In addition, operation with a gas is only possible to a very limited extent (septa). Theoretically, operation under vacuum would only be possible to a very limited extent. The septa are no longer leak-tight, at the latest after each penetration by the needles usually employed. In addition, a part of the evaporated medium, for example, condenses on the cold septa which, on the one hand, makes contamination of the reaction mixture probable due, for example, to softeners in the plastic of the septum (this also applies to all the other applications of septa in the chemical or biological reaction procedure described here). In addition, however, particularly in association with the sealing argument quoted above, very many applications demand evaporation of high-boiling-point solvents, for example dimethyl formamide or dimethyl sulphoxide, in a relatively short time and/or at relatively low temperatures (due, for example, to the usually limited stability of chemical compounds in chemical or biological processes). The reaction vessels cannot be fully opened and closed automatically.

A reaction vessel holding device is known from US-A-5 503 805 which has a fixed part and a multiplicity of reaction vessels fastened to it by means of flexible hoses. The supply of liquid into the reaction vessels takes place from a starting vessel via a further hose, a multiple valve and the flexible hoses mentioned or otherwise from below. One disadvantage of this device consists in the fact that powder cannot be supplied to the reaction vessels, that it is not possible to dose precisely in this way and that the supply of different liquids in different reaction vessels is relatively complicated because the supply takes place in each case via the starting vessel or otherwise from below.

In view of the disadvantages of the previously known devices, as described above, the invention is based on the following object. A reaction vessel holding device of the type mentioned at the beginning is to be created which permits a movement of reaction vessels fastened to it, without this movement being transmitted to the fixed part, and which permits a supply of liquid, solids and gas into the reaction vessels and an extraction from the reaction vessels in a simple manner.

This object is achieved by means of the reaction vessel holding device according to the invention, as defined in the independent Claim 1. A reaction block according to the invention with such a reaction vessel holding device is defined in Claim 12. Preferred embodiment variants are given by the dependent claims.

The essential feature of the invention consists

in the fact that in a reaction vessel holding device having a fixed part and a plurality of connectors, which are connected to it and to which reaction vessels can be fastened, at least one part of each connector being flexible, at least one of the connectors and the fixed part have an opening through which a supply and/or extraction tool can be introduced into a reaction vessel fastened to the connector.

Because the reaction vessels are fastened by means of movable, and advantageously removable, connectors on the fixed part, which fixed part can be configured, for example, as a switching block or other block with or without clamped, screwed or inserted septa, the reaction vessels can be shaken without the relevant fixed part moving with them. By means of additional axial flexibility of the flexible connectors, it is even possible for at least two reaction vessels which are rigidly connected together to be shaken. In addition, the full weight of the reaction vessels, including content, is not laid on the shaking device so that relatively weak shaking devices are sufficient to generate the vibrations, which are relatively small despite relatively large shaking amplitudes. Only this arrangement makes it at all possible to use vibration-sensitive peripheral units, such for example as samplers, robots or other automation devices for the supply and/or extraction of liquids and/or solids. Furthermore, such relatively weak shaking devices have favourable costs and the reaction vessels can be shaken with a relatively large amplitude at very high frequencies.

An essential and decisive advantage lies in the fact that, even during the shaking, it is possible to

supply and/or extract a liquid, a gas or a solid to or from a reaction vessel without difficulty by means of a supply and/or extraction tool, such for example as hollow needles, grippers or spoons. This is not the case with the previously known devices but is frequently demanded, particularly for chemical reactions.

Such connectors advantageously have a bellows between a rigid fastening part at the reaction vessel end and a part at the fixed part end. Alternatives to the bellows are, for example, a flexible tube, a spherical joint or a twin-axis or multiple-axis linkage.

Liquids, gases and/or solids can preferably be supplied and/or extracted to or from the reaction vessels through the fixed part. If the fixed part is configured as a switching block, it advantageously comprises a gas duct plate, a gas duct and, advantageously, an adjacent functional plate, one of these plates being advantageously arranged so that it can be displaced relative to the other. The gas duct plate or plates and the functional plate or plates have through-holes, through-slots and/or depressions and/or horizontal through-cavities, which are located opposite one another, in each case in at least one plate position, in such a way that

a) gases and/or liquids can be supplied to or extracted from at least one reaction vessel via the gas duct or via at least one gas duct, and

b) gases, liquids and/or solids can be supplied to or extracted from at least one reaction vessel both through the gas duct plate or through at least one gas duct plate and through the functional plate or through at least one functional plate.

By means of this switching block, it is possible - depending on the plate position - to supply or extract liquids or gases to or from all or individual reaction vessels via the gas duct or ducts and/or to generate a vacuum or a positive pressure in the reaction vessels and/or to supply or extract gases, liquids and/or solids to or from the reaction vessels with or without pressure balance through mutually opposite through-holes or through-slots with holes and/or depressions with holes and/or through-cavities with holes in the gas duct plate or plates and in the functional plate or plates. The sealing of the reaction vessels takes place by means of the functional plate or by means of at least one functional plate in certain plate positions so that septa are not absolutely necessary. It is therefore possible to dispense with the use of a septum or it can, if necessary, be arranged in such a way that it is only used as a seal for the reaction vessels in certain plate positions.

If, instead of being attached to a switching block, the flexible connectors are attached to a simple block as the fixed part, the same advantages as described above exist, with the exception of those which are specifically associated with the functional plate.

Because of the flexible connectors and the functional plate or plates, various process steps can be carried out without the relevant switching block having to be displaced or modified. The functional plate or plates also permit a compact design of the switching blocks.

For operating the switching blocks or blocks with or without clamped, screwed or inserted septa and

the reaction vessels, a commercially obtainable sampler combined with a dilutor, for example the Gilson Aspec XL of the Gilson company, France, or another robot or another automatic unit can be employed, which units may possibly be adapted to the switching blocks according to the invention or to blocks with or without clamped, screwed or inserted septa and reaction vessels. As mentioned above, the flexible connectors can also, however, be attached - with all the advantages described above - to a simpler block provided with little switching or none at all. Thus, for example, the dosing described here is likewise possible during the shaking and, in fact, as well if septa are employed as if the reaction vessels are left open. Pressure balance is, for example, ensured by means of the needle (commercially obtainable) during the supply of liquid or by means of a central pipe with a permanent or a (for example by means of a valve) switchable connection to, for example, an argon source and, for example, permanent connections of the central gas duct to the through-holes, i.e. to the reaction vessels.

For certain applications, a removable reflux cooler is preferably arranged between at least one of the reaction vessels and the fixed part. This reflux cooler has a cooling tube which extends sufficiently far into the reaction vessel for cooling to take place in the reaction vessel in the connection region between the reaction vessel and the reflux cooler or below this region. It is then advantageous for the cooling tube to be arranged in one half only of the opening cross section of the reaction vessel, so that the supply and extraction instrument can be centrally immersed deep

into the reaction vessel.

In this way, it is possible - with simultaneous reflux cooling - to supply a liquid to the reaction vessel, for example by means of a supply tool or via the or a gas duct, to add and/or extract a protective gas, gaseous reactant, gaseous catalyst or a solid, and/or a pressure balance can be achieved and/or the reaction vessels can be shaken at high frequency. All these interventions in the reaction vessel take place through the same opening, which contributes to the fact that the device according to the invention can be constructed in a relatively compact and low-cost manner and be maintenance-friendly and operator-friendly.

For further applications, special reaction vessels are necessary which permit the reaction vessel, or its contents, to be tempered over a temperature range which is as wide as possible. The reaction vessels produced for this purpose advantageously have a second chamber, which is fitted in a fixed or removable manner at the lower end of the reaction vessel and through which a tempering medium can be pumped. Units for delivering tempered media are obtainable on the market - a Unistat of Huber GmbH, which can be employed over a very wide temperature range, can, for example, be used. This unit, together with a special medium (for example polydimethyl siloxane, No. 85413 from Fluka AG) permits tempering of the reaction vessel in the range from approximately -80°C to approximately +200°C. So that these reaction vessels can be shaken in the manner described above (i.e. while using flexible connectors, i.e. with simultaneous dosing of a material, for example), a decisive feature is that the second cham-

ber, which is firmly connected to the reaction vessel, should be directly or indirectly flexibly connected to the tempering unit and/or to the further reaction vessels (connected, for example, in parallel) and/or to another medium-conveying unit. An example of an indirect connection between two reaction vessels is shown in Fig. 26. In this arrangement, a second chamber of one reaction vessel is connected via a helical, medium-carrying line to a second chamber of another reaction vessel. The simplest arrangement is for flexible hoses composed of plastic to be employed for connecting the tempering chambers to one another and to the tempering unit. Since, particularly in the case of applications which demand a very large temperature range (-80 to +300°C), plastics either have too little flexibility (particularly at low temperatures) and/or are excessively unstable (particularly at high temperatures), specially produced and arranged helical connectors in form of hollow metal wires (for example aluminium, copper, steel, spring steel) or Teflon hoses (most of the commercially available fluorinated hydrocarbon polymers are suitable) are advantageously employed as the connections between the tempering chambers and the tempering unit, particularly in the case of dense packings of reaction vessels with, therefore, small distances between the tempering chambers. As an alternative to this system, flexible chambers with connecting sleeves for the reaction vessels have been specially produced. These chambers are produced in enriched form, for example, from silicone of various hardness, thermoplastic elastomers, such as, for example, polyethylene or polypropylene, copolymers of, for

example, styrene, ethylene or butylene with silicone oil. These flexible chambers satisfy the same conditions as the chambers composed of a stiff material described above, which are flexibly connected together or to the tempering unit by means of medium-carrying lines. In this case, the flexibility is achieved by means of the flexible material from which the chambers are manufactured instead of by means of hoses composed of a flexible material or by means of the sprung helices composed of a relatively stiff material. In principle, the chambers correspond to a "hot-water bottle", with the reaction vessels advantageously let into them in such a way that the latter are in direct contact with the heat carrier and are sealed toward the outside so that the heat carrier remains in the circuit.

Reaction vessels, which permit, for example, a mixture of an insoluble and a soluble material to be separated by filtration, are useful and often necessary for further applications. This advantageously takes place, by means of the arrangement according to the invention, in such a way that the filtrate is transferred without manual intervention for further processing into a second reaction vessel, so that it is available for further processing (for example distilling off the solvent) on the same access plane of, for example, the sampler employed. The same advantages appear in the case of the filter cake present in the reaction vessel before the frit. This is achieved by means of the reaction vessels described in Fig. 20, together with a special function in the fixed part, or with analogous valve controls, in such a way that the reaction vessel, which contains the frit, is closed in a vacuum-tight

manner and, at the same time, the reaction vessel, which catches the filtrate, is placed under vacuum. The same can be achieved by pressure instead of vacuum so that - expressing the matter generally - a relative positive pressure has to be generated in the reaction vessel with the frit. The switching block described above automatically makes this necessary pressure difference possible, controlled by means of a PC, because the displaceable plate takes up the corresponding asymmetrical position. All the twin filtration vessels connected, or the mixtures located in them, are then filtered in parallel.

All the possible combinations can also be employed with all the reaction vessels mentioned above for the special and further applications quoted. As an example, the reflux coolers can be placed on the twin filtration vessels, which are in turn provided with the second chambers, described above, for tempering the contents of the reaction vessels. One or a plurality of these combinations can then, for example, be placed on a switching block or a simple block with flexible connectors which are attached in a fixed or removable manner. By means of embodiment examples of flexible connectors, which are also axially flexible, it is also possible for each two rigidly connected reaction vessels to be shaken with the advantages described, i.e. with simultaneous supply, for example, of a material by means of a dosing device.

The reaction vessel holding device according to the invention and the reaction block according to the invention are now described in more detail with reference to the attached drawings. In these:

Fig. 1 shows a perspective view, obliquely from above, of an embodiment example of a device for carrying out a multiplicity of chemical, biochemical, biological or physical processes in parallel;

Fig. 2 shows a perspective view, obliquely from below, of an embodiment example of a device for carrying out a multiplicity of chemical, biochemical, biological or physical processes in parallel, a flexible connector being also shown;

Fig. 3 shows a side view of a connector;

Fig. 4 shows a section through the connector of Fig. 3 along the line A-A;

Fig. 5 shows a simple block with a gas duct, with septum plate omitted and cover plate omitted, so that the connections of the reaction vessels via depressions in the block and the connections to the gas duct are visible, with two embodiment examples of flexible connectors;

Fig. 6 shows a perspective view of an embodiment example of a simple block with individual septa screwed on and two rows of three reaction vessels each with indicated shaking device and connection of the reaction vessels to the same;

Fig. 7 shows a switching block with an embodiment example of a flexible connector

and an embodiment example of a reaction vessel attached to it;

Fig. 8 shows the switching block of Fig. 7 with two different embodiment examples of a flexible connector broken down into its individual parts;

Fig. 9 shows an embodiment example of the bottom of the functional plate of the switching block of Fig. 7;

Fig. 10 shows an embodiment example of the top of the functional plate of the switching block of Fig. 7;

Fig. 11 shows a segment of an embodiment variant of a slide plate of the switching block of Fig. 7, the necessary connections between the reaction vessels and the gas duct of the gas duct plate being configured as through-cavities;

Figures 12 to 15 show sections through the segment of Fig. 11, which show the construction of the through-cavities;

Fig. 16 shows a perspective view of the gas duct plate of the switching block of Fig. 7;

Fig. 17 shows a side view of the gas duct plate of the switching block of Fig. 7, parts which are not visible being shown dotted;

Fig. 18 shows a plan view of the gas duct plate of the switching block of Fig. 7, parts which are not visible

being shown dotted;

Fig. 19 shows a flexible connector with a simple reaction vessel fastened to it and an embodiment example of a reflux cooler;

Fig. 20 shows an embodiment example of two reaction vessels connected flexibly or rigidly to a tube, with a frit in one of the two reaction vessels in order to permit filtration, for example, of a mixture into the second reaction vessel, which is located at the same level;

Figures 21, 22 show an embodiment example of a reaction vessel with a second attached chamber, with connections for a supply line and a drain line, in order to permit a series or parallel connection of the reaction vessels arranged in parallel to one another or to a tempering unit, in order to permit tempering of the reaction mixture;

Fig. 23 shows a section through the reaction vessel of Fig. 22 along the line B-B;

Fig. 24 shows a section through the reaction vessel of Fig. 22 along the line C-C;

Fig. 25 shows a section through the reaction vessel of Fig. 22 along the line D-D;

Fig. 26 shows an embodiment example of two reaction vessels flexibly connected to one another in the most constricted space below the flexible part of the

connector;

Fig. 27a shows a flexible chamber composed of a material which is stable and flexible over a large temperature range and in which the reaction vessels are immersed, so that a lower part of the latter is directly in contact with the tempering medium and an emergence of tempering medium from the chamber is prevented by sealing lips;

Fig. 27b shows a section through the flexible chamber of Fig. 27a;

Fig. 28 shows a flexible chamber in accordance with Fig. 27a, which includes a central part of the reaction vessel; and

Fig. 29 shows a diagrammatic circuit diagram of a device according to the invention for carrying out a multiplicity of chemical, biochemical, biological or physical processes in parallel.

Fig. 1

The embodiment example shown, of a device for carrying out a multiplicity of chemical, biochemical, biological or physical processes in parallel, comprises a support frame 24, into which two switching blocks 1 are inserted and between which there is still space for three further switching blocks 1. The switching blocks 1 are fastened by means of screws, for which the support frame 24 has screw holes 241. An educt vessel frame 25, which is used for holding educt vessels 250, is also arranged on the support frame 24. The educt

vessel frame 25 is configured in such a way that the educt vessels 250 can be arranged on two planes. This serves the better utilization of the space available and therefore serves to increase the number of educt vessels 250. Two holding blocks or capture plates 27 are provided for holding additional educt vessels 250 or sample extraction vessels. Solvent extraction points 28 permit the extraction of solvents from solvent tanks.

An arm 26 of a sampler is used to support a hollow needle for the handling of starting materials or products. The corners of the access surface for the hollow needles 261 are indicated by needles 261.

A shaking device 7, a vacuum pump 2, a plurality of gas supply devices, valves for the gas supply devices and the vacuum pump, tempering units, a plurality of control units, a dilutor and a plurality of reaction vessels 5 are also part of the device, and are shown further below in Figure 29.

The following statement applies to the further description overall. If reference numbers are contained in a figure for the purpose of drawing clarity but are not mentioned in the directly associated descriptive text, reference is to be made to their mention in the previous description of figures.

Fig. 2

An embodiment variant of the device for carrying out a multiplicity of chemical, biochemical, biological or physical processes in parallel, from Fig. 1, has two flexible connectors 4 and another frame 25 for holding educts.

Figures 3 and 4

The connector 4 shown comprises a part 41 at the block end and a part 42 at the reaction vessel end, between which is arranged a bellows 43 which satisfies the function that the part 42 at the reaction vessel end should be movable relative to the part 41 at the block end and, in fact, both laterally and also in the x-, y- and z-direction. The part 41 at the block end is provided with a thread 411 so that the connector 4 can be screwed into a hole 111 of the switching block, which hole 111 is shown in Fig. 16 and is provided with an internal thread. The part 42 at the reaction vessel end additionally comprises a fixing region 421 for applying fastening clamps and a standard ground joint 422 for the releasable fastening of a reaction vessel 5 or other parts used, such as, for example, reflux cooler 6. The connector 4 has a central opening 433.

Fig. 5

A simple block 1' comprises a gas duct plate 11' and two embodiment examples of flexible connectors 4, 4' - one with a flexible bellows 43, the other with a flexible tube 43' - as the flexible part of the connector. A rigid fastening part 42 or 42', respectively, is attached to the flexible part for fastening a reaction vessel 5.

The connector 4, which is described in more detail below, is screwed by means of a thread 411 into the bottom of the block shown, into an internal thread of the block. The opposite end is used for the removable fastening of reaction vessels 5. In

addition, the reaction vessels can be sealed by means of a septum plate, which is not visible here.

The connector 4 can, for example, consist of the materials which are described further below in association with Fig. 7.

Gas duct holes are indicated by 113, through-holes by 114, screw holes by 161, a hole for a pin by 162 and depressions by 119 (see also Fig. 8 and 16-18).

Fig. 6

A section through an embodiment example of a block 1 with a row consisting of three reaction vessels, which are each firmly sealed on the block 1 by a septum 73 (fixing by means of plastic screw caps 74). The reaction vessels 5 are respectively connected to the switching block 1 by means of a flexible connector 4. The reaction vessels 5 are guided by a drive 71 which is firmly connected to the diagrammatically shown shaker 7. The shaking motion takes place in the direction of the arrow. The co-ordinate axes 58 indicate that the end 59 of the reaction vessels facing away from the connector and the end 57 of the reaction vessels at the connector side can be moved in three degrees of freedom.

Fig. 7

The flexible connectors 4, 4' described in Fig. 5 are used for the removable fastening of reaction vessels 5 on the switching block 1. Each through-hole 111 of the gas duct plate can be associated with a reaction vessel 5 or a plurality of holes 75 can be associated with a reaction vessel 5.

A switching block 1 used in the device of Fig. 1 has a gas duct plate 11, a functional plate 12 in the form of a slide plate, a backing plate 13 and a support plate 14, which are located one above the other. The support plate 14, the backing plate 13 and the gas duct plate 11 are held together by screws, which are arranged in screw holes 10. An end plate 16 with holes for pins and screw holes 161 additionally connects these three plates by means of pins and screws (not shown). The functional plate 12 is arranged so that it can be displaced between the gas duct plate 11 and the backing plate 13. It is driven by a stepper motor 15 via a pinion 151 and a rack 152. Fastening, distance and positioning elements are indicated by the reference number 18.

The support plate 14 also has two rows of through-holes 131, which permit a needle 261, described in Fig. 1, or some other solid object to pass through the plate. This plate is mainly used for stabilization, for protection and for holding the drive device for the functional plate 12. A panel 135 is used as a collecting point for the light barrier signals. As an alternative to this arrangement, a standard interface can be used for controlling the functions listed above.

The backing plate 13 has screw holes 10' and through-holes 10, which are located opposite the screw holes 10' and through-holes 10 of the support plate 14. It preferably consists of a high-quality material and, in particular, its side facing toward the functional plate 12 is made more exactly than the support plate 14.

The functional plate 14 is narrower than the

backing plate 13 and the gas duct plate 11, so that it fits exactly between the connecting screws of these two plates and is also even guided by these connecting screws. It includes through-holes 10, whose rims are slightly raised relative to the plate surface and thus ensure a good seal. On the bottom, furthermore, it also has depressions which are explained in more detail further below.

Fig. 8

In addition to the screw holes 114 and the through-holes 10, the gas duct plate 11 has gas duct holes 113, which end in a central gas duct 112. An appropriate number of vacuum pumps and gas supply devices can be connected to the open end of the gas duct 112 by means of a valve, preferably a multiple valve. These vacuum pumps and gas supply devices supply the reaction vessels 5 via the flexible connectors 4'.

Between the backing plate 13 and the support plate 14, it is also possible to arrange a septum composed of a material which can be penetrated by a needle, which septum acts as an additional, optional seal for the reaction vessel openings when the through-holes of the backing plate 13, the gas duct plate 11 and the functional plate 12 are located opposite one another.

The individual parts of the switching block can, for example, consist of metal (in particular stainless steel, brass or titanium alloys), glass (in particular SiO_2 glass), plastic (in particular Teflon, polypropylene or polyethylene), natural stone (in

particular granite or gneis), or ceramic (in particular AlO_3 or MACOR®). The connectors can, for example, consist of plastic or a metal, in particular of Teflon, polypropylene, polyethylene or steel sheet.

Fig. 9

The bottom of the functional plate 12 has, in this case, a pattern which is repeated eight times. An individual pattern comprises four different arrangements of through-holes 121, depressions 122 and sealing surfaces 123 which, depending on the plate position, are located opposite the through-holes 121 and the gas duct holes 113 of the gas duct plate 11 and/or of the support plate 13 and, in this way, define four different functional plate functions.

The first arrangement has two through-holes 121 and, between the latter, a depression 122. The first functional plate function therefore leaves both associated reaction vessels 5 completely open, i.e. both for supply and/or extraction tools and relative to the gas duct. A single long depression 12 is present in the case of the second arrangement.

The third arrangement comprises a depression and two sealing surfaces 123. The third functional plate function completely closes off an associated reaction vessel, whereas it leaves the other associated reaction vessel 5 open relative to the gas duct 112 only, so that a pressure difference can be generated in rows of reaction vessels connected in parallel.

In the fourth arrangement, two through-holes 121 and two depressions 123 are present. The fourth functional plate function leaves the two associated

reaction vessels 5 open for supply and/or extraction tools but closes them off relative to the gas duct 112.

The rims of the through-holes 121, the depressions 122 and the sealing surfaces 123 are all slightly raised above the plate surface and therefore ensure a good seal, with the possibility of a complete sealing of the reaction vessels 5 or the possibility of applying a vacuum.

Other arrangements of the through-holes 121, the depressions 122 and the sealing surfaces 123, and consequently other functional plate functions or other patterns, are of course also conceivable.

Fig. 10

This functional plate 12' differs from the functional plate 12 in that, on the top, it is not just the rims of the through-holes 121 which are raised above the plate surface but also the regions between the through-holes 121.

Fig. 11

An alternative embodiment variant of the functional plate 12 described in Fig. 7, only one segment of the pattern, which is repeated eight times, of a complete functional plate 12 being represented. The slots shown in Fig. 7 are configured as through-cavities 129 in this embodiment example, the individual positions of the functional plate being represented in the sequence of Fig. 12 to Fig. 15. Otherwise, the same functionality is provided.

Figures 12 to 15

Sections which represent the individual positions of the functional plate in the sequence Fig. 12 to Fig. 15.

Figures 16, 17 and 18

In order to ensure sufficient drainage of possibly condensed solvents as far as the closed end, the gas duct plate 11 shown has a central gas duct which rises slightly from the open end and from which gas-duct holes 114 extend to the plate surface facing toward the functional plate. The through-holes 113 are arranged in two parallel rows corresponding to the through-holes 132 of the backing plate 13 and the through-holes of the support plate 14 and the screw holes 141 are arranged to correspond respectively to the screw holes 131 or those of these plates. Screw holes for fastening the end plate are indicated by 161. A multiple valve, to which an appropriate number of vacuum pumps and/or devices for the supply of one or a plurality of gases is connected, is advantageously attached, for example screwed in, at the open end of the gas duct 11. A vacuum or a positive pressure can then be generated in the reaction vessels 5 and/or the most varied gases can be supplied to the reaction vessels 5. In this way, the atmospheres and conditions achievable by means of the functional plate functions can be multiplied, with simultaneous shaking, in the reaction vessels 5.

Fig. 19

In its upper region, the reflux cooler 6 shown

has a standard ground joint inner surface 62 for the releasable fastening of the reflux cooler 6 to the standard ground joint 63 of a connector 4 or a reaction vessel 5 and, in its lower region, it has a standard ground joint external surface 63 for the releasable fastening of a reaction vessel 5. It also includes a cooling tube 61, which extends sufficiently far into the reaction vessel 5 for cooling to take place in the reaction vessel 5 below the connection region 51 of the reaction vessel 5 and the reflux cooler 6. This achieves the effect that the gas phase condenses relatively far down in the reaction vessel; the condensate therefore remains in the reaction vessel 5 and condenses before the standard ground joint connection 62.

The cooling tube 61 is arranged asymmetrically in the opening cross section of the reaction vessel 5, i.e. it is displaced toward the outside relative to the centre of the reflux cooler in order to create space for the introduction into the reaction vessel 5 of a supply and/or extraction tool or the addition of, for example, a protective gas, gaseous reactant, gaseous catalyst or a solid, etc. The supply and removal of the cooling medium, for example water, takes place as shown by the arrows B and C by means of flexible supply and removal lines, which are arranged and are connected, for example, to the supply and removal lines of the reflux cooler of further reaction vessels in such a way that their space requirement is minimized.

Fig. 20

In the device according to the invention, many different types of reaction vessels can, in principle,

be used; they all, however, have a connection possibility, such as a standard ground joint 62 for example, for the releasable or firm fastening of the reaction vessel to, for example, a reflux cooler 6 or a connector 4. The shape and the acceptable volumes of the reaction vessels can be varied over a wide range as a function of the available space and the desired number of reaction vessels to be used adjacent to one another. As an example, cylindrical reaction vessels with round or flat bottoms, round beakers, pointed beakers, etc, in particular with acceptable volumes between 0.3 ml - 200 ml, can be considered.

Two reaction vessels 5" and 5"', which can be used for filtration, and which are connected by a flexible or a rigid line 52, are shown here. The first end of the tube protrudes into the upper region of the reaction vessel 5", whereas the second end is melted into a frit 53, for example glass frit, in the bottom region of the reaction vessel 5"'. Filtration can be carried out through the frit 53 by the generation of pressure in the reaction vessel 5"', as shown by the arrow, and/or by the generation of vacuum in the reaction vessel 5", as shown by the arrow, and this can take place with simultaneous and/or previous shaking and, in fact, in such a way that both the filter cake remaining in front of the frit 53 and the filtrate are available at the same level for access by the supply device for further processing.

<u>Figures 21 to 25</u>

It is also possible to use reaction vessels 5' onto which an additional chamber 8 with an inlet and an

outlet 81, 81', 81" and 81"' is melted. These chambers can be used as cooling or heating chambers and are preferably connected to one another in a space-saving manner. The reaction vessels 5 described in Fig. 12 can also be provided with additional cooling or heating chambers and/or can be combined with reflux coolers 6. Fundamentally, the most widely varying combinations of these elements are conceivable with all possible types of reaction vessels 5.

Fig. 26

An alternative reaction vessel 5" with a second additional chamber 8 with connections for the supply and removal lines 81, 81' and, attached to them, lines 82, 82', 82" for media, which lines - in order to build up, in the most restricted space, mechanical flexibility between the two reaction vessels 5" adjacent to one another - are wound in the form of helices around a rod 84 or 84' attached to the two chambers 8.

On the left-hand reaction vessel, a reflux cooler 6 with the reflux cooler tube 61 is attached by means of the standard ground joint 63 to the reaction vessel 5", which is in turn connected by means of the standard ground joint 62 to the flexible connector 4.

Figures 27a and 27b

An embodiment example of a flexible chamber 9 composed of a flexible material which is stable over a large temperature range (plastics such as silicone, relatively thin-walled Teflon, polypropylene, etc), in which the reaction vessels 5 are immersed in such a way that a lower part of the reaction vessel 5 is in direct

contact with the tempering medium. The outlet of tempering medium from the chamber is achieved by means of the upwardly extended cylindrical walls 91 and the sealing rings 92. The supply of the heat carrier takes place by means of the connections 93.

Fig. 28

An alternative variant of a flexible chamber 9 differs from that described in Figures 27a and 27b in that the reaction vessel not only enters the flexible chamber but passes through the latter so that the tempering medium is in direct contact with a central region of the reaction vessel 5. In consequence, upwardly extended cylindrical walls 91 and sealing rings 92 are fitted both at the inlet point and at the outlet point. The connections 93 for the supply lines are fitted at the end surface.

Fig. 29

The present embodiment example of a device has five switching blocks 1, below which a shaking device 7 is arranged. The access to the gas duct 112 of the respective gas duct plate 11 is controlled by multiple valves 3, by means of which a vacuum can be generated by a vacuum pump 23 or else gas can be supplied or extracted by means of a device for the supply or extraction of gas. In the embodiment example shown, the gas supply and extraction device includes a tank for a gaseous material, for example argon, hydrogen, etc.

At least one cryostatic temperature regulator is provided to supply each of the cooling tubes 61 of the reflux cooler 6 and each of the melted-on, second

additional chambers 8 or the flexible chambers 9 of the reaction vessels 5 with a heat carrier. The handling of the starting materials or products takes place by means of a device for the supply and extraction of liquids and/or solids, which device comprises one or a plurality of hollow needles 261 or other supply and/or extraction tools. The starting materials or products are stored, in part, in vessels 250 which are arranged in the vessel frame 25. Solvent or solution extraction points 28 permit the extraction of solvents or solutions from tanks.

A control unit 88, for example a PC, is used for controlling the supply and extraction of liquids, gases and/or solids, i.e. for controlling a device 261 for the supply and extraction of liquids and/or solids, for controlling the device for the supply and extraction of gases, for controlling the vacuum pump 23, for controlling the multiple valves 3 and for controlling the functional plates 9 and their motors 15, and for controlling the shaking device 7 and the cryostatic temperature regulators 29, 89.

In addition to the previously described device for carrying out a multiplicity of chemical, biochemical, biological or physical processes in parallel, further design variations can be realized.

It is expressly mentioned at this point that the connections between the connectors 4, the reflux coolers 6 and the reaction vessels 5 do not necessarily have to take place by means of standard ground joints 63 but, for example, threads, face grinding, etc can also be provided. Similarly, the connection 411 between the block and the flexible connector 4 does not

necessarily have to be screwed but can be a fixed connection, a ground face or a bayonet fixing.

Claims

1. Reaction vessel holding device, having a fixed part (1; 1') and a plurality of connectors (4, 4') which are connected to it and to which reaction vessels (5; 5'; 5"; 5"') can be fastened, at least one part of each connector (4, 4') being flexible, characterized in that at least one of the connectors (4, 4') and the fixed part (1; 1') have an opening (433, 111, 121) through which a supply and/or extraction tool can be introduced into a reaction vessel (5; 5'; 5"; 5"') fastened to the connector (4, 4').

2. Reaction vessel holding device according to Claim 1, characterized in that the supply and/or extraction tool is a needle (261), a spoon or a gripper.

3. Reaction vessel holding device according to Claim 1 or 2, characterized in that at least one connector (4, 4') is configured in such a way that a reaction vessel (5; 5'; 5"; 5"') fastened to the connector (4, 4') can be shaken without the fixed part (1; 1') being moved.

4. Reaction vessel holding device according to Claim 3, characterized in that each of the connectors (4, 4') is configured in such a way that each reaction vessel (5; 5'; 5"; 5"') fastened to one of the connectors (4, 4') can be shaken without the fixed part (1; 1') being moved.

5. Reaction vessel holding device according to one of Claims 1 to 4, characterized in that at least one connector (4, 4') is configured in such a way that the end (59) facing away from the connector, of a reaction vessel (5) fastened to the connector (4, 4') can be moved in three degrees of freedom.

6. Reaction vessel holding device according to Claim 5, characterized in that at least one connector (4, 4') is configured in such a way that the end (57), on the connector side, of a reaction vessel (5) fastened to the connector (4, 4') can be moved in three degrees of freedom.

7. Reaction vessel holding device according to one of Claims 1 to 6, characterized in that at least one connector (4, 4') is configured in such a way that it ensures a vacuum-tight and pressure-tight connection between the reaction vessel (5; 5'; 5"; 5"') and the fixed part (1; 1').

8. Reaction vessel holding device according to one of Claims 1 to 7, characterized in that at least one connector (4, 4') has, at the reaction vessel end, a rigid fastening part (42; 42') which is attached to a flexible part, in particular a bellows (43), a flexible tube (43') or a spherical joint.

9. Reaction vessel holding device according to one of Claims 1 to 8, characterized in that a septum (73) is arranged over or in the fixed part (1), in at least one connector or between at least one connector

and the fixed part.

10. Reaction vessel holding device according to one of Claims 1 to 9, characterized in that the fixed part is configured as a switching block (1) of a device for carrying out a multiplicity of chemical, biochemical, biological or physical processes in parallel.

11. Reaction vessel holding device according to Claim 10, characterized in that the device for carrying out a multiplicity of chemical, biochemical, biological or physical processes in parallel has at least one switching block (1) to which a multiplicity of reaction vessels (5; 5'; 5"; 5"') can be fastened in a removable or fixed manner, it being possible to supply and/or extract liquids, gases and/or solids to or from the reaction vessels (5; 5'; 5"; 5"') through the switching block (1), the switching block (1) comprising at least one gas duct plate (11) with at least one gas duct (112) and at least one adjacent functional plate (12), at least one of these plates being arranged so that it can be displaced relative to the other and the gas duct plate (11) or plates and the functional plate (12) or plates having through-holes (111, 121), through-slots, depressions (122) and/or through-cavities (129) which are located opposite one another, in each case in at least one plate position, in such a way that

a) gases and/or liquids can be supplied to or extracted from at least one reaction vessel (5; 5'; 5"; 5"') via the gas duct or via at least one gas duct (112), and

b) gases, liquids and/or solids can be supplied to or extracted from at least one reaction vessel (5; 5'; 5"; 5"') both through the gas duct plate (11) or through at least one gas duct plate and through the functional plate (12) or through at least one functional plate.

12. Reaction block, characterized in that it comprises a reaction vessel holding device according to one of Claims 1 to 11 with a plurality of reaction vessels (5; 5'; 5"; 5"') fastened to it.

13. Reaction block according to Claim 12, characterized in that a reflux cooler (6) is arranged between the reaction vessel holding device and at least one reaction vessel (5; 5"), which reflux cooler (6) has a cooling tube (61) which extends sufficiently far into the reaction vessel (5; 5") for cooling to take place in the reaction vessel (5; 5") in the connection region between the reaction vessel (5; 5") and the reflux cooler (6) or below this region, it being possible to arrange the cooling tube (61), in particular, in one half only of the opening cross section of the reaction vessel (5; 5").

14. Reaction block according to Claim 12 or 13, characterized in that at least two reaction vessels (5; 5") are flexibly connected directly or indirectly to one another and/or to a tempering unit (29, 89) and/or another medium-carrying unit by means of a medium-carrying line (82, 82', 82"; 82"') or chamber (9).

15. Reaction block according to Claim 14, characterized in that the flexibility of the medium-carrying line (82, 82', 82"; 82"') is achieved by a helical arrangement.

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Patent Agents

FIG. 1

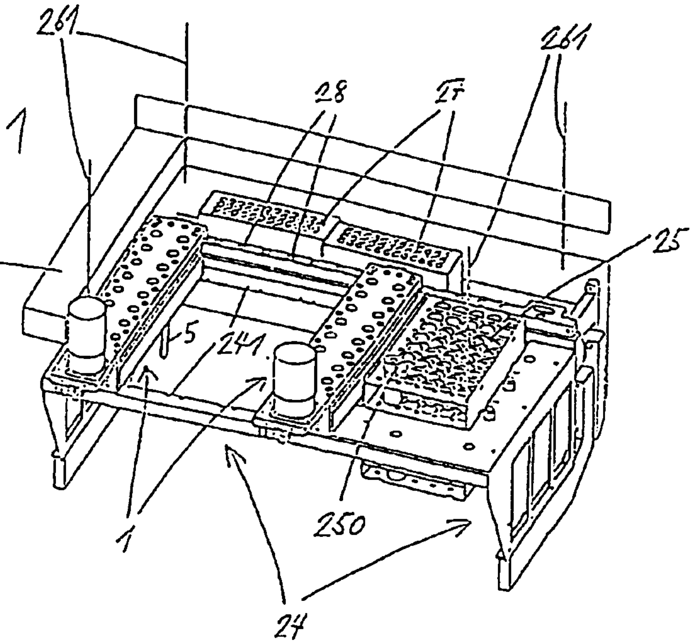


FIG. 2

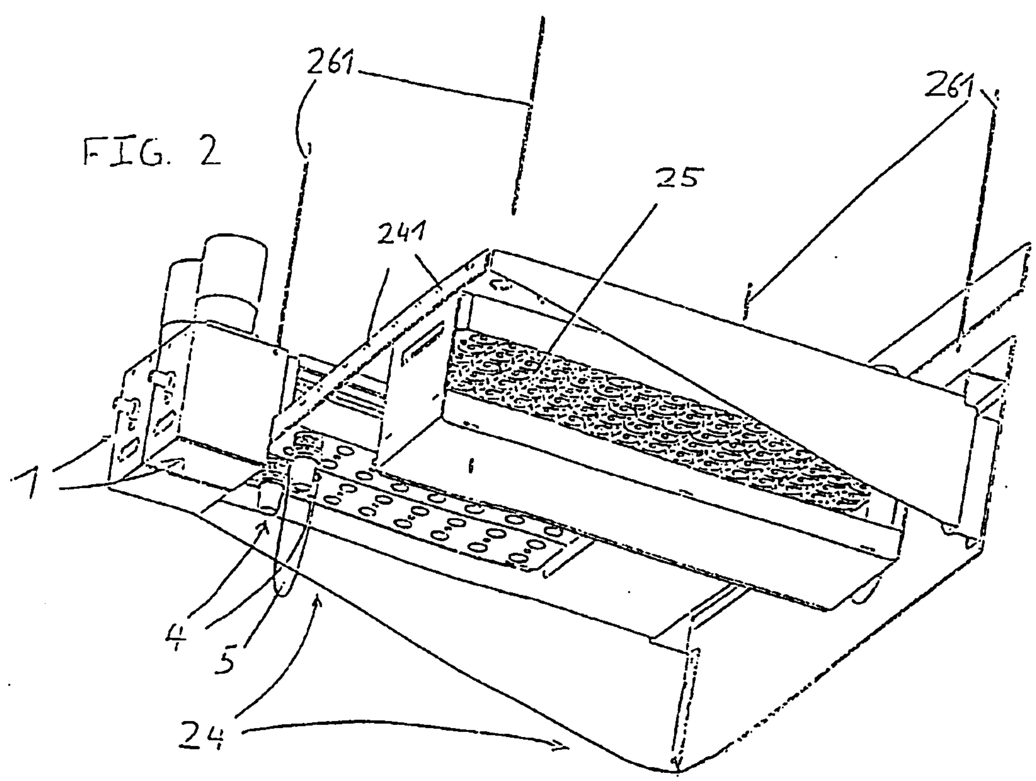


FIG.3

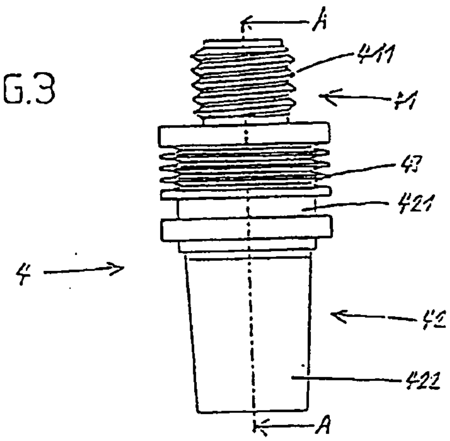


FIG.4

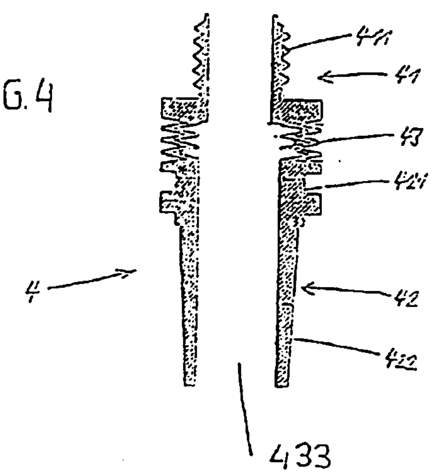


FIG. 5

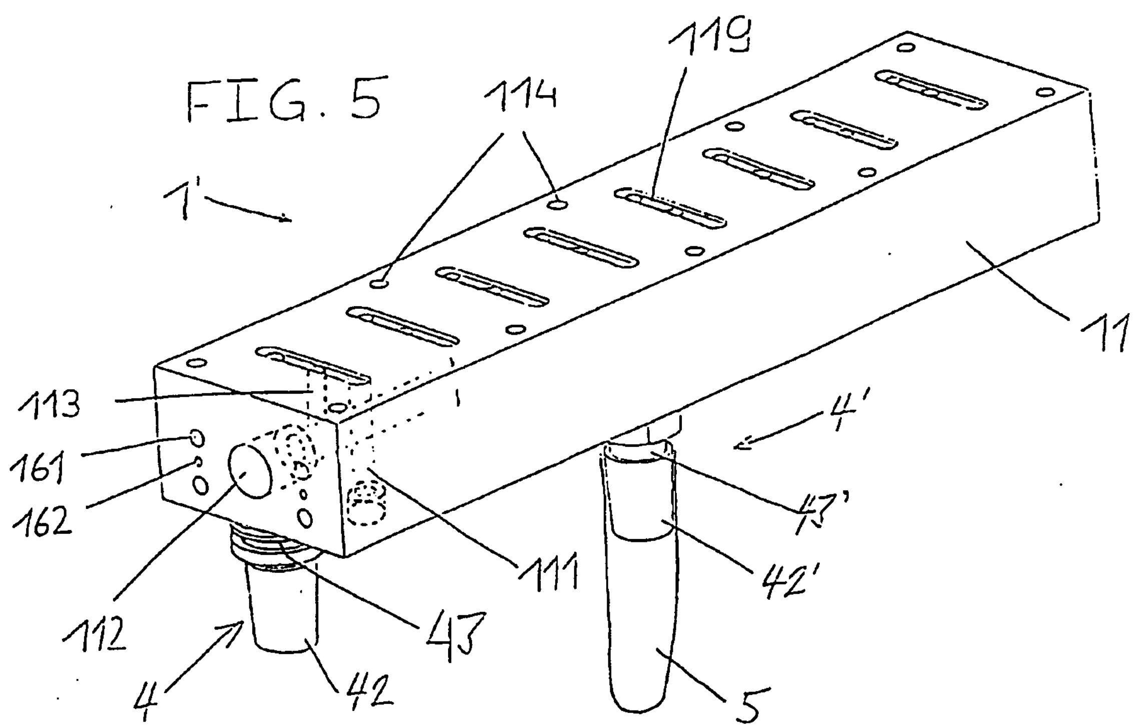


FIG.6

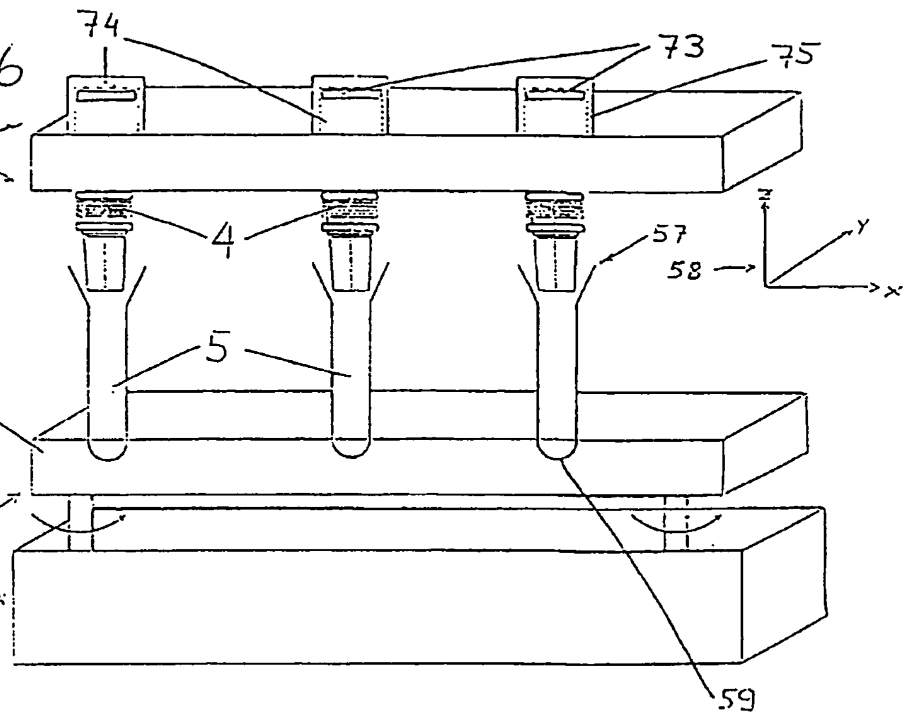

74
73
75
1
4
57
58
z
y
x
5
71
7
59

FIG.7

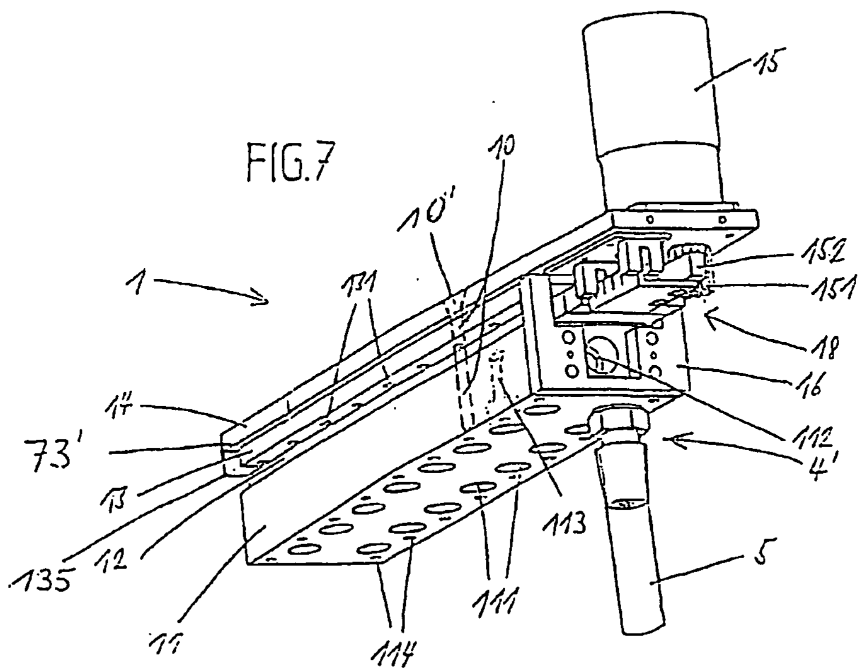

15
10
10'
1
131
152
151
18
16
14
73'
112
4'
13
113
5
135
12
111
11
114

FIG. 8

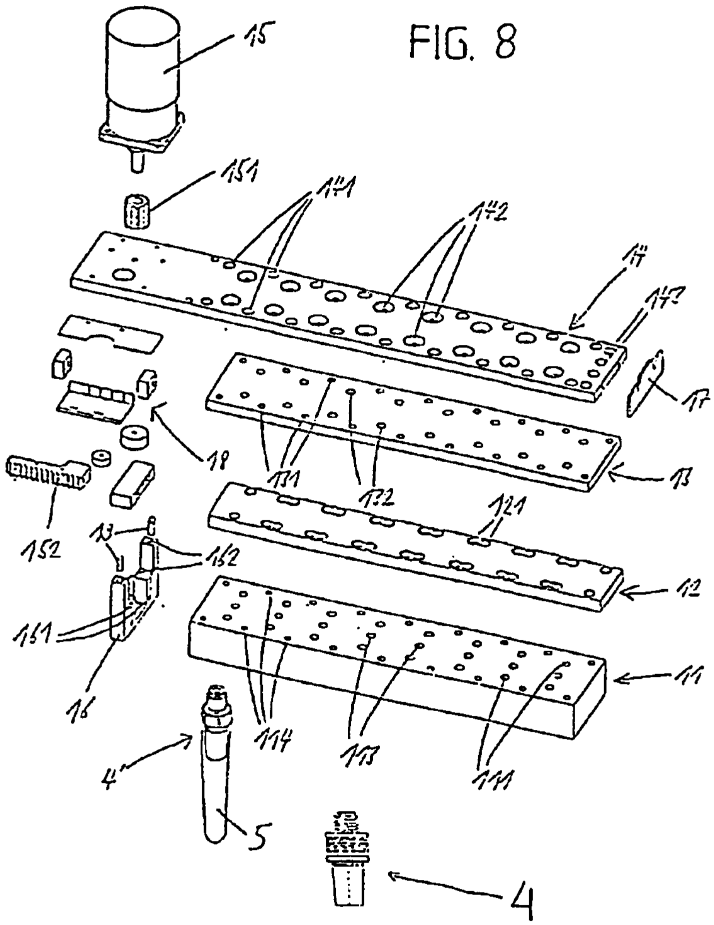


FIG. 9

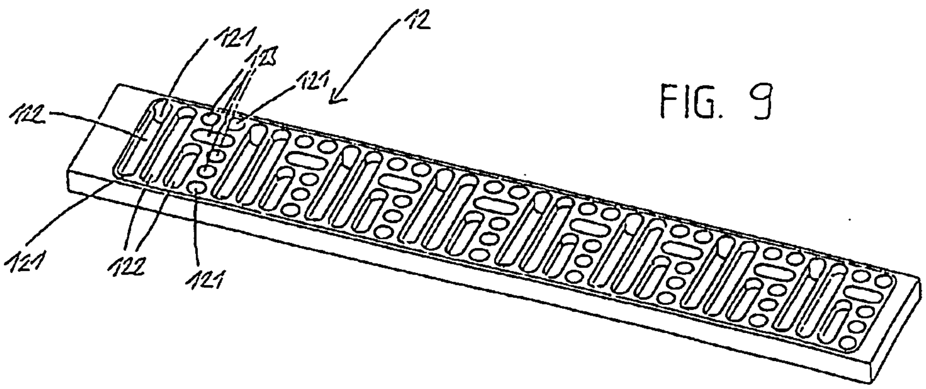
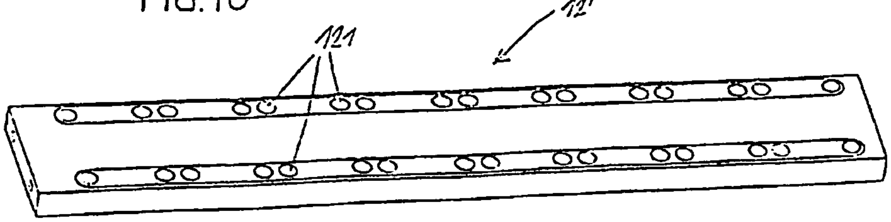
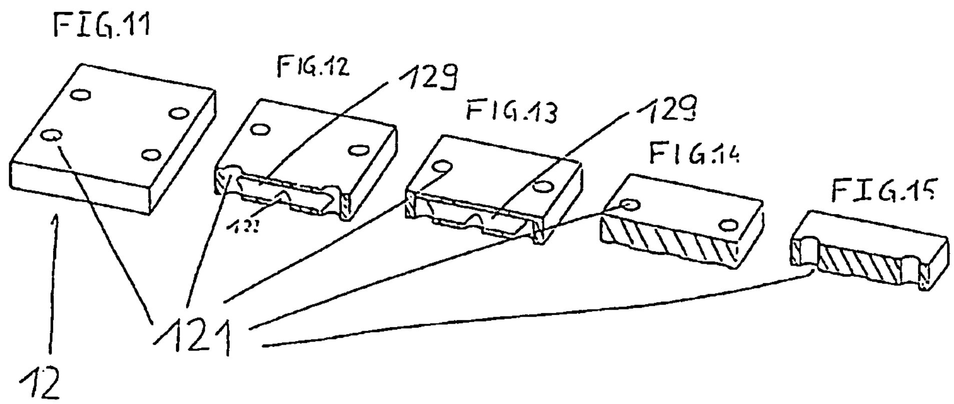


FIG.10





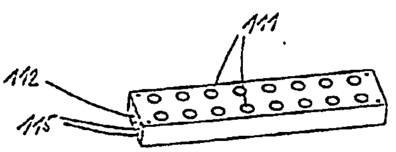


FIG.16

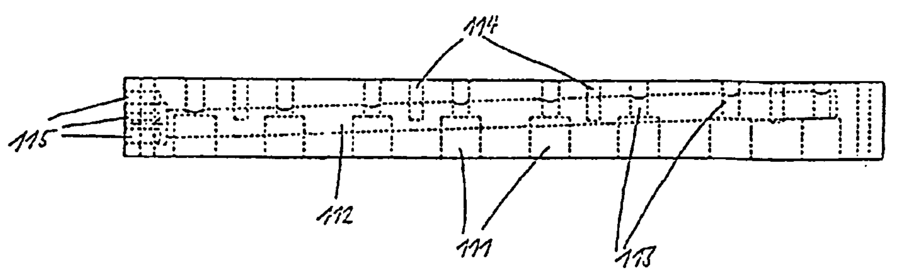


FIG.17

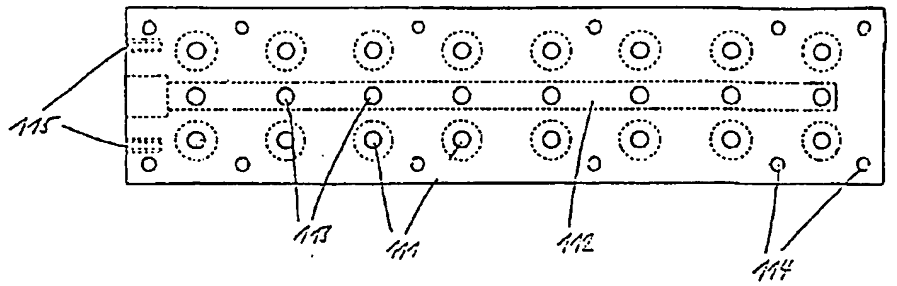


FIG. 18

FIG. 19

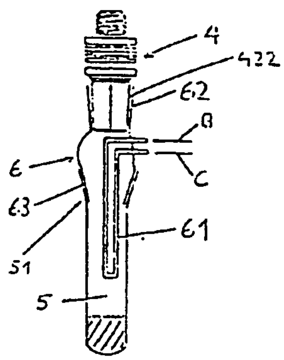


FIG. 20

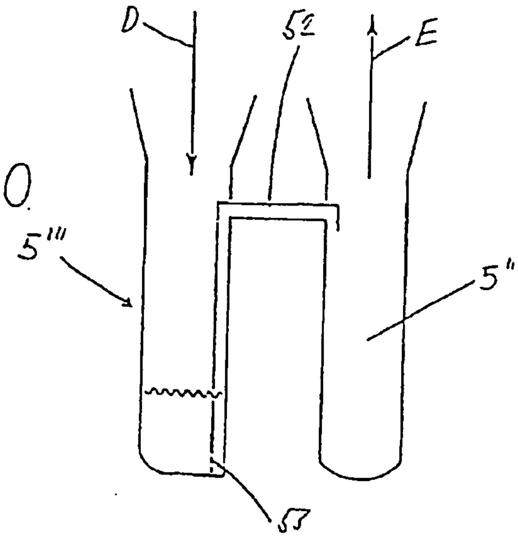


FIG. 22

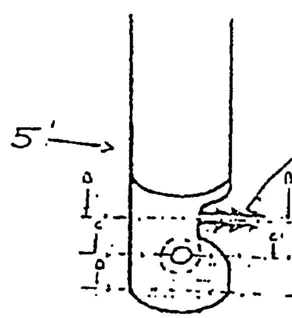


FIG. 21

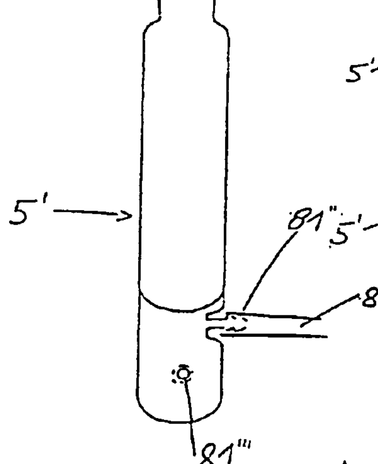


FIG. 23

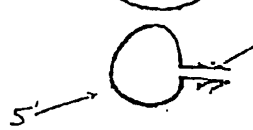


FIG. 24

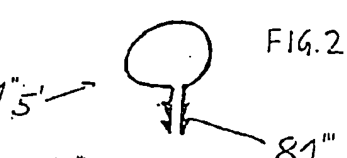


FIG. 25

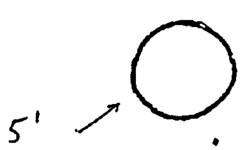


FIG.26

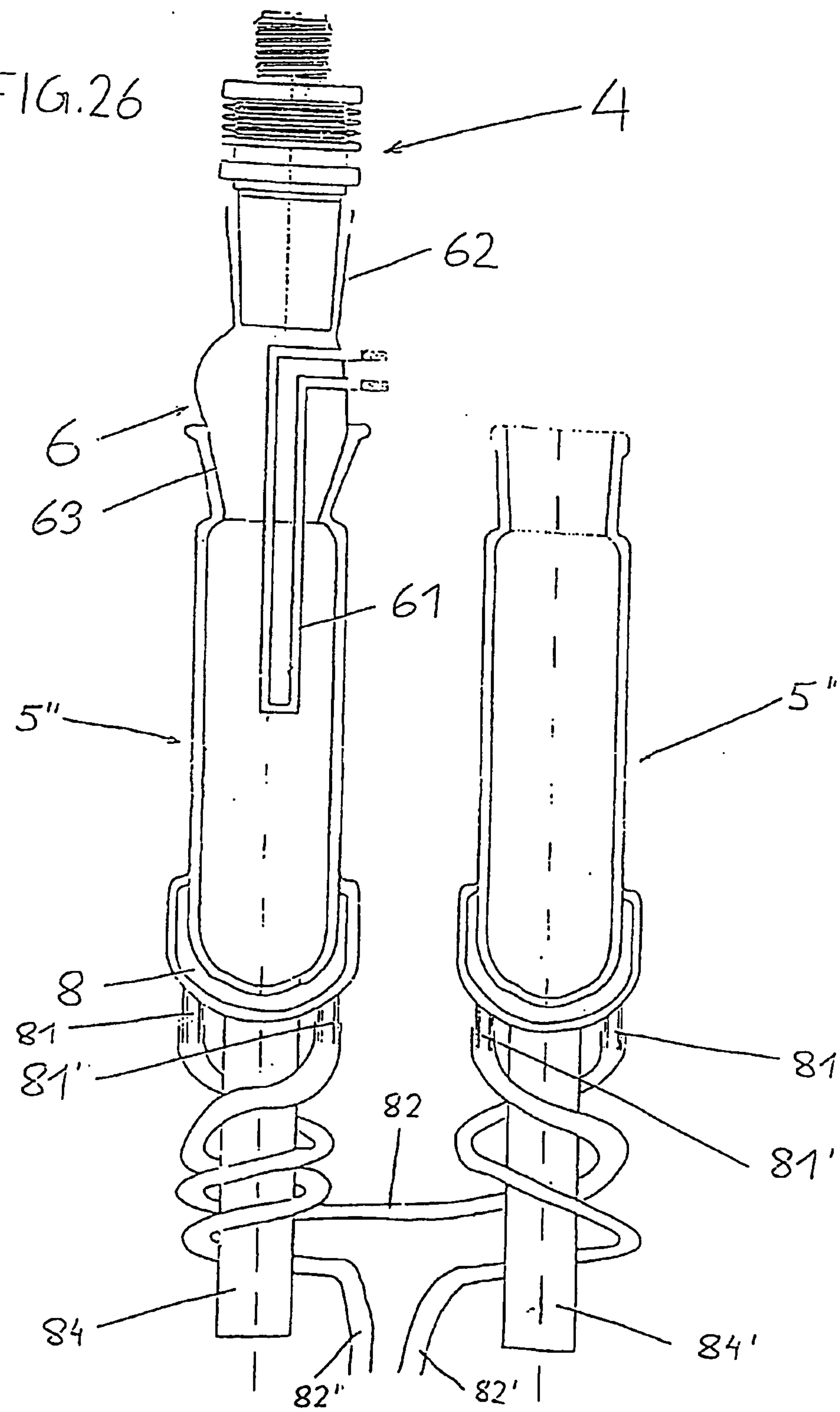


FIG. 27a

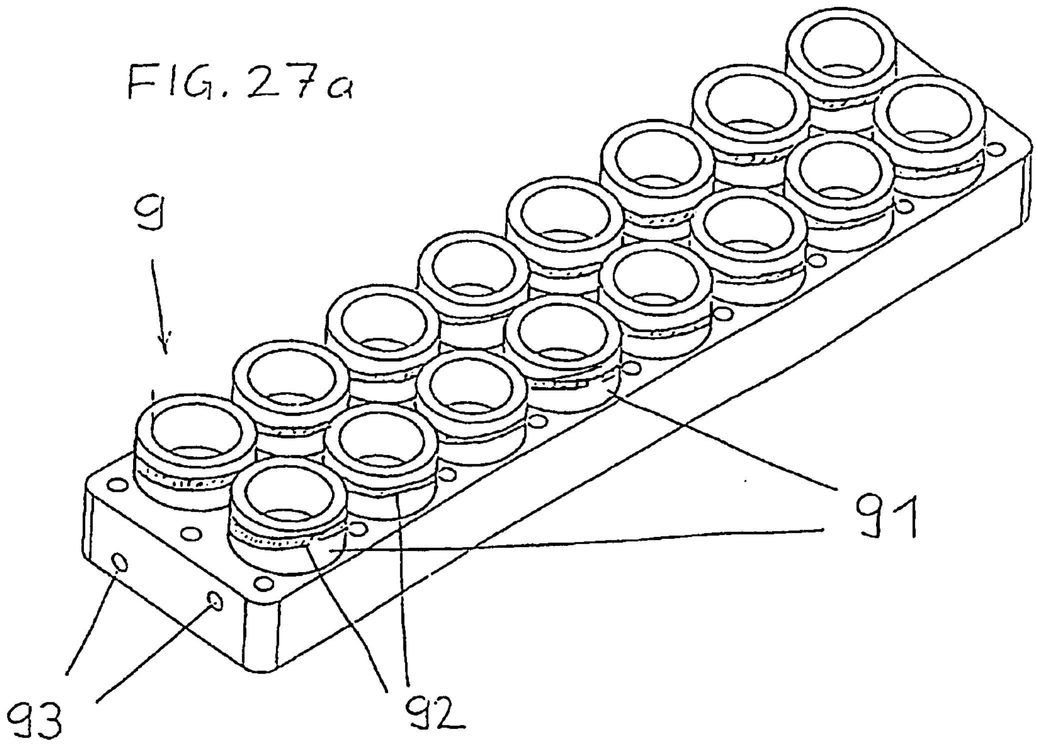


FIG. 27b

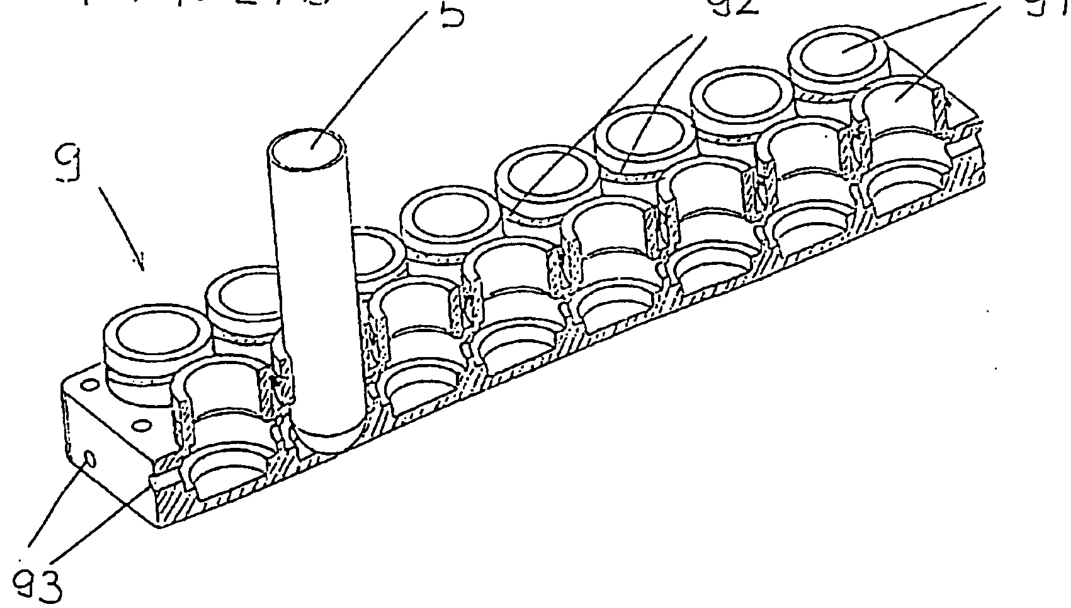


FIG.28

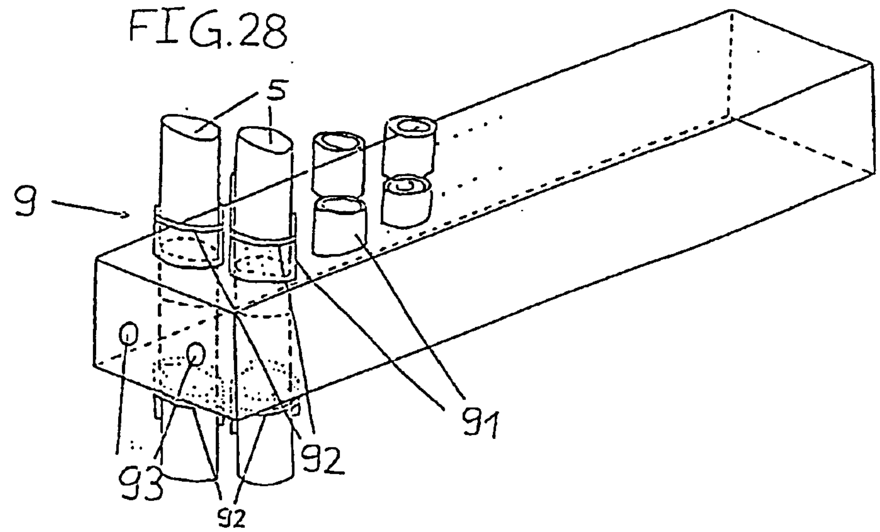


FIG.29